The Effect of Samples Geometry and Thermal Cycling Test Type on the Thermal Shock Behaviour of Plasma Sprayed TBCs

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Thermal barrier coatings (TBCs) are effective in protecting gas turbine blades by insulating them from hot gas passing through. In addition, they provide prevention of thermal shock, oxidation resistance and improved engine efficiency. A major life-limiting weakness of TBCs is their susceptibility to get damaged by thermally-induced stresses. The thermal shock behaviour of TBC systems subjected to thermal shock loading and the influence of the cracks on the coating durability was investigated experimentally. In this study, TBCs (NiCrAlY + YSZ), which have different sample geometry were subjected to both of gas burner and furnace thermal cycle tests. Sample geometry and cycle test type effect on the correlation of thermal shock behaviour of TBC coatings were characterized by macroscopic and microstructural investigations.

Introduction

The demands for increased engine efficiency and performance in terms of higher operating temperatures and lower emissions continue to serve as the driving force for the advance of thermal barrier coating (TBC) technology. Plasma sprayed TBCs are widely used in hot section components of gas turbines to increase gas turbine inlet temperature, thus increasing the efficiency and performance of the engine.[1–3]

Thermal barrier coatings have a tendency to spall, or debond, under cyclic high temperature conditions. It is believed that spallation of the ceramic component in TBC system is a result of the stresses generated in service.[4] The performance of TBC system is also affected by thermal expansion mismatch between the ceramic and the metal, thermal stresses generated by the temperature gradients in the TBC system.[5] In this study, two thermal cycling tests are presented, which simulate the turbine operating conditions and allow a wide range of thermal gradients. The aim of this study was to examine the effect of samples geometry and thermal cycle test type on the thermal shock behaviour of plasma sprayed YSZ coatings.

Experimental Part

Materials and Coating Process

Thermal barrier coatings, comprising a bond coat and a top coat, were sprayed onto an AISI 310S (X12CrNi2521) heat resistant stainless steel substrate. Two different substrates geometry were chosen for the investigation of thermal shock behaviour of the TBCs. One of them was a disc-shaped plate, diameter of 25.4 mm and thickness of 3 mm, being flat in the middle and rounded with a well-defined radius of curvature of 1.5 mm at the perimeter, as shown in Figure 1. This geometry reduced residual stress originating at the free edge of the samples. The other specimen geometry was a square of 30 × 30 mm² surface area with 3 mm thickness.

The spraying materials for the bond coat and the top coat were NiCrAlY (Ni17Cr-12Al-1Y (in wt. %)) and yttria stabilized zirconia
(YSZ: ZrO$_2$-8 wt.-% Y$_2$O$_3$), respectively. Before the deposition process, the substrates were grit blasted and subsequently placed in an air plasma spray (APS) system for overlaying with the NiCrAlY bond coating ($\sim$120–150 $\mu$m). ZrO$_2$–Y$_2$O$_3$ was sprayed onto as sprayed bond coat specimen in an APS Ar/H$_2$ system using 3MB gun. The top coat thicknesses are varied 180–200 $\mu$m. The parameters of the coating process are given in Table 1.

**Thermal Cycle Tests**

Two different types of thermal cycle tests were performed.

(1) **Furnace Test:** The samples were heated in the tube furnace (Figure 2a). The samples were pushed into the furnace when the temperature of the furnace reached up to 1000 $^\circ$C. They were held for about 300 s in the furnace at 1000 $^\circ$C and then quenched by compressed air. The cooling time for air quenching was about 120 s. More than 20% of spalled region of the surface of the top coating was adopted as criteria for the failure of the coating in air quenched specimens. This type of thermal shock testing was also performed by other investigators.[$^5$–$^{11}$]

(2) **Gas Burner Test:** Thermal cycling was performed in two gas burner test facilities operating with propane and oxygen (Figure 2b). The substrates were cooled by compressed air from the back to provide temperature gradient. The surface temperature was measured with a pyrometer. The substrate temperature was measured by a thermocouple mounted in a hole drilled 15 mm towards the centre of the substrate from one side. The surface temperature was varied between about 1240 and 1320 $^\circ$C, and the substrate temperature was adjusted between 960 and 1020 $^\circ$C. In the test facilities, gas burners with a broad flame were used to give a fairly homogeneous temperature distribution in the centre of the samples. After being heated for about 20 s, the maximum temperature was nearly reached. After 5 min, the burner was automatically removed for 2 min from the surface, and the surface was cooled from the front at an initial rate of about 100 K s$^{-1}$ using compressed air. The cycles were carried out for 5 min heating and 2 min cooling, and it was repeated until failure occurred. Cycling was stopped when a visible spallation of the coating occurred[$^{12}$–$^{14}$]

**Characterization**

Coated samples were impregnated with epoxy and then sectioned, ground and polished. The surface planar section and cross-section of the coatings were examined macroscopically by using scanning electron microscopy (SEM).

**Results**

**Microstructure**

Cross-sectional SEM micrograph of the as-sprayed coating are shown in Figure 3. The coatings consist of a ceramic based top coat on a metallic bond coat and metallic substrate. Plasma sprayed zirconia is a lamellar structure made of mechanically interlocking molten splats and has highly micro scaled cracks and porosities. Microcracks and pores could be easily observed from this microstructure. However, it was not examined to check if the cracks were distinctively vertical or horizontal. Additionally, unmelted particles were also observed in the bond coat.

**Thermal Shock Behaviour of TBC**

Thermal shock resistance is related to the size and distribution of pores or existing cracks in the coating. When TBC is used cyclically from high temperature to low temperature, cracking and spalling occurs. Cyclic application of thermal loads can cause horizontal or vertical cracks to propagate resulting in delamination/spallation of the
coating and loss of thermal protection to the substrate.\textsuperscript{[15]} When the spallation area reaches about 5\% of total coating area, the TBC sample is considered as failure. The number of thermal cycles recorded is an indication of thermal shock resistance.\textsuperscript{[16]}

The macro images of the surfaces of the two different geometry coated samples, after gas burner test, are shown in Figure 4. Gas burner test creates extreme temperature gradients in ceramic layer. As can be seen from here, flame was focussed to the centre of samples, and thus flame ashes can be seen on images. The failure, which occurred on the disc-shaped specimen, appeared after 200 cycles. Chipping was observed as coating failure mechanism starting from the edge of specimen (Figure 4c). From macro images, delamination and buckling type failures were not examined for the disc-shaped sample. The first failure on square shaped sample was observed when cycle number reached 100 (Figure 4e). On one edge of square, spallation and delamination failures occurred. Additionally, bending and distortion of substrate were observed for square shaped sample, and not observed for disc-shaped substrate.

Macroscopic images of two different samples after furnace cycling test are given in Figure 5. The failure of disc-shaped sample occurred when cycle number reached 368. Delamination area was indicated by 1 number arrow. In this area, bond coat was peeled because of severe oxidation (Figure 5c). The top coating, which is indicated by 2 number arrow, delaminated between top and bond coat interface. Quite a number of segmentation cracks were present in the coating, most of which went through the coating thickness, for both of the different shaped samples. Although it can be seen that there is sample bending failure for square shaped sample, there is not any bending or distortion for disc-shaped sample.

The first failure initiated from two edges of square at 100 cycle of testing and peeling of the coating appeared severely chipped at 268 cycles (Figure 5e–f). The failure took place between bond coat and top coat. As can be seen from Figure 5f, there is some residual ceramic of top coat on the failure area.

Scanning electron microscopy investigation showed that two different cracks were inspected from surface of the coating, depending on thermal cycle test type. It can be seen from Figure 6a that the crack initiated in the centre of flame area of disc-shaped samples and propagated three different directions in the burner test. Additionally, the flame contact area cracked partially. After furnace testing, segmentation cracks network was observed on sample surface. Both cases include top surface investigations after 100 cycles.

As a result of the cross-section SEM investigations, horizontal cracks were observed throughout thickness in gas burner test (Figure 7a). Otherwise vertical cracks were observed after furnace tests. In the furnace test, vertical cracks joined with horizontal cracks (Figure 7b). As a result of this, surface spalling occurred (Figure 5f).

**Discussion**

In gas burner test, TBC system is exposed to dynamically thermal cycles and thermal gradient is formed between coating and substrate.\textsuperscript{[12]} During the cycles, the plane tensile stresses were generated due to the thermal expansion difference between the coating and substrate.\textsuperscript{[13–15]} Horizontal cracks occurred near the top coat/bond coat interface with increase in
the cycling number. The horizontal cracks within the top coat propagate and coalesce with thermal cycling. When a crack with sufficient size was reached, the ceramic layer separated from the substrate, and edge chipping occurred. In both type of samples, the thermal cycle chipping occurred in the edges of sample. If the surface of sample is exposed directly to burner flame, which has high heat flux, an elastic module of the central surface increases by sintering. The sintering shrinkage in the coating provides compressive stresses to decrease during thermal cycling. Thus upon cooling, the surface of the coating becomes tensed, and cracks consequently. Macroscopically, there is no delamination or spallation on disc shaped samples surface after thermal cycles. On the other hand, top coat delamination was observed on square shaped samples. It is observed that failure origins from sample edges. However, there is no spallation experienced on flame areas of the square shaped samples.

Thermal gradients were not formed in furnace tests. Disc shaped samples were tested by this thermal cycle test method and similar delamination failures, which originated from the sample edges, were observed. Macroscopically, there were no segmentation crack networks in the centre of the sample surfaces. No deformation was observed on disc shaped samples. It can be seen that besides coating spallations in the middle region of the specimen, extensive coating delaminations and spallations also occurred near the specimen edges because the exposure of the entire specimen during the furnace thermal cycles resulted in severe edge stress concentration, and thus initiated edge cracking.

As expected, in the square sample, failure originated on the edges and corners and depending on the numbers of thermal cycles spallation was observed on the whole surface of the sample. In addition, test sample plastically deformed and bended. As a result of this, delamination and spallation failures happened. The test results are summarized in Table 2.

In electron microscopic studies, it is observed that failures which occurred on surface were established by different mechanisms. To simulate the thermal shock phenomenon accurately, appointed factors should be carefully considered, such as sample geometry and test type. It is thought that sample geometry affects thermal shock failure type. Figure 6a shows an SEM micrograph of a topcoat after gas burner test. It is observed that in the image taken from flame area, the crack origins from the centre and it moves along three different directions. And also, there is a partial mass loss in the central area of the flame after 100 cycles. After progressing cycles, surface deteriorated and segmentation crack networks were observed.

Cross-sectional SEM micrographs of the samples showed that dissimilarity of test method affect the thermal cycle failure type. In gas burner tested samples, cracks included the horizontal types. These cracks induced lamellar
separation from surface and consequently delamination occurred. In furnace tests, vertical cracks in the top coat combined with horizontal cracks and local spallation came about similarly for early researchers as well. Delamination emerged between bond coat and top coat. Cooling down to room temperature develops compressive stresses in the coating. As the stress level increases beyond a critical value, it can finally lead to a spallation of the layers, as observed in the experiments. The cracks propagate and coalesce with the increase in thermal cycling. Results show that the thermal gradient affects the surface and interface cracking.

Conclusions

The influence of sample geometry (disc and square) and thermal cycle test type (furnace test and gas burner test) on the thermal shock behaviour of plasma sprayed TBCs were investigated. Results are given below.

Disc-shaped specimens showed that delaminating and spalling failure under furnace thermal cycle testing, while square shaped specimen showed edge chipping and specimen bending. In the gas burner thermal cycling test system, square shaped specimens showed delamination at edge and distortion failure between bond coat interfaces. For disc-shaped specimen, failure appeared as chipping in the ceramic top coat.

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Table 2. Thermal cycle test results.

<table>
<thead>
<tr>
<th>Substrate geometry</th>
<th>Furnace test</th>
<th>Gas burner test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Failure area</td>
<td>Top coat</td>
<td>Bond coat</td>
</tr>
<tr>
<td>Failure type</td>
<td>Edge chipping</td>
<td>Delamination spalling</td>
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<tr>
<td>Cycle numbers</td>
<td>268</td>
<td>368 100 200</td>
</tr>
</tbody>
</table>

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